भारतीय मानक
Indian Standard

IS 16873 : 2018

केस्सिया गम, खाद्य ग्रेड — विशिष्टि

Cassia Gum, Food Grade — Specification

ICS 67.220.20

© BIS 2018

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली–110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.org.in www.standardsbis.in

September 2018 **Price Group 3**

Food Additives Sectional Committee, FAD 08

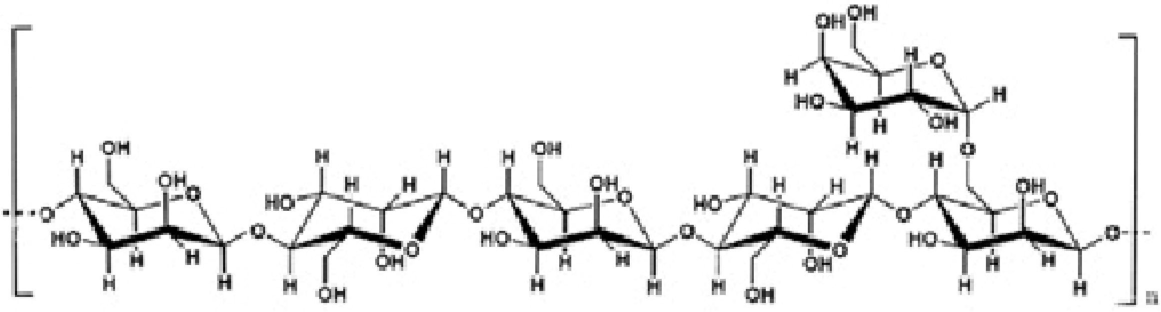
FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

Cassia gum is a food additive made from the endosperm of *Cassia obtusifolia* also known as *Senna obtusifolia* or *Cassia tora*. Cassia gum is a naturally occurring polymer with mannose and galactose as its repeating units. Cassia gum forms high viscosity by swelling in water after it is boiled. Cassia gum is the purified flour from the endosperm of the seeds of *Cassia obtusifolia* which belongs to the leguminosae family. The seeds that are used in the production of cassia gum are generally picked randomly from these seeds. Before the seed splitting process is started, the seeds of *Cassia obtusifolia* are passed through grading machines to differentiate them on the basis of their size. During this procedure, the underdeveloped seeds are removed. The seeds are dehusked and de-germed by thermal mechanical treatment followed by milling and screening of the endosperm. The ground endosperm is further purified by extraction with isopropanol. Cassia gum is used as thickener, emulsifier, foam stabilizer, moisture retention agent and texturizing agent in various food products.

Cassia is a tonic, carminative and stimulant. It is permitted as a food additive under the *Food Safety and Standards (Food Products Standards and Food Additives) Regulations*, 2011.

Structural formula:



In the formulation of this standard the requirements given in the following publications have been taken into consideration:

a) Codex General Standards for Food Additives (GSFA) FAO JECFA Monographs 10 (2010), and
b) E 427 under the European Commission Directive 2010/67/EU of 20 October 2010.

In the formulation of this standard, due consideration has been given to the provisions of the *Food Safety and Standards Act*, 2006 and the *Rules* and *Regulations* framed thereunder and the *Legal Metrology (Packaged Commodities) Rules*, 2011. However, this standard is subject to the restrictions imposed under these, wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

CASSIA GUM, FOOD GRADE — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for Cassia gum, food grade.

2 REFERENCES

The standards given below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of these standards indicated below :

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
1699 : 1994	Methods of sampling and test for food colours
2491 : 2013	Food hygiene — General principles — Code of practice
5402 : 2012 / ISO 4833 : 2003	Microbiology of food and animal feeding stuffs — Horizontal method for the enumeration of micro-organisms — Colony count technique at 30°C by pour plate technique (*second revision*)
5887	Methods for detection of bacteria responsible for food poisoning:
(Part 1) : 1976	Isolation, identification and enumeration of *Escherichia coli* (*first revision*)
(Part 3) : 1999	General guidance on methods for detection of Salmonella (*second revision*)
7219 : 1973	Method for determination of proteins in foods and feed ingredients
7928 : 1992	Alginic acid, food grade (*first revision*)
13360 (Part 11/ Sec 10) : 1999	Plastics — Methods of testing: Part 11 Special properties, Section 10 resins in the liquid state or as emulsions or dispersions — Determination of apparent viscosity by the Brookfield test method
16069 (Part 2) : 2013	Microbiology of food and animal feeding stuffs — Horizontal method for the enumeration of yeasts and moulds : Part 2 colony count technique in products with water activity less than or equal to 0.95
IS/ISO 6492 : 1999	Animal feeding stuffs — Determination of fat content

3 REQUIREMENTS

3.1 Description

Cassia gum shall be off white to yellowish free flowing powder. It shall be free from dirt, dust, added colouring matter, visible mould growth, insect infestation, black splits, stems, straw, chaff and obnoxious smell.

3.2 Identification

3.2.1 Cassia gum shall be insoluble in ethanol. It shall disperse well in cold water forming a colloidal solution.

3.2.2 An aqueous dispersion of Cassia gum having *p*H above 9.0 shall be converted to a gel by the addition of small amounts of 2 percent sodium borate solution.

3.2.3 The product shall pass the test for formation of gel with xanthan gum as per the procedure given in Annex A.

3.2.4 When tested as per method given in Annex B the product shall indicate the presence of mannose and galactose.

3.2.5 No blue colour shall be produced when a few drops of iodine solution is added to a 1 in 10 aqueous dispersion of the product.

3.3 Hygienic Conditions

The product shall be manufactured and packed under hygienic conditions (*see* IS 2491).

3.4 Microbiological Requirements

3.4.1 *Escherichia coli* shall be absent in 1g of the sample when tested in accordance with the method given in IS 5887 (Part 1).

3.4.2 *Salmonella* shall be absent in 25g sample when tested in accordance with the method given in IS 5887 (Part 3).

3.4.3 *Yeast and mould* shall not exceed 100/g when tested in accordance with the method given in IS 16069 (Part 2).

3.4.4 The total viable colony count shall not exceed 5 000/g when tested in accordance with the methods given in IS 5402.

3.5 Residual isopropanol shall not be more than 0.75 percent by mass when tested as per the method given in Annex E.

3.6 The material shall also conform to the requirements given in Table 1.

4 PACKING, STORAGE AND MARKING

4.1 Packing

The material shall be packed only in sound clean, dry and un-used containers made of B-twill jute or in polythene bags placed in gunny bags or multi-ply craft paper sacks or any other material as agreed to between the purchaser and supplier. The material shall be filled in containers with as little air space as possible. The containers shall be such as to preclude air contamination of the contents with metals or other impurities.

4.2 Storage

The material shall be stored in a cool and dry place so as to avoid excessive exposure to heat.

4.3 Marking

Each container shall be marked legibly to give the following information:

a) Name of the material including the words 'Food Grade';
b) Source of manufacture;
c) Net Quantity when packed;
d) Batch or code number;
e) Date of manufacture;
f) Instruction for storage;
g) Best before date (month and year to be given by the manufacturer); and
h) Any other requirements as specified under the *Legal Metrology (Packaged Commodities) Rules,* 2011 and *Food Safety and Standards (Packaging and Labelling) Regulation*, 2011 and the Rules framed thereunder.

4.4 BIS Certification Marking

The container may also be marked with the Standard Mark.

4.4.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

The representative samples of the material shall be drawn and conformity of the material to the requirements of this specification shall be determined according to the procedure prescribed in **4** of IS 1699.

6 TESTS

6.1 Tests shall be carried out by the methods specified in **3.2**, **3.4** and col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE—'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

Table 1 Requirements for Cassia Gum, Food Grade

(*Clause* 3.6)

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Galactomannans, percent by mass, *Min*	75.0	Annex C
ii)	Loss on drying, percent by mass, *Max*	12.0	**A-2** of IS 7928
iii)	Total ash, percent by mass, *Max*	1.2	**A-4** of IS 7928
iv)	Acid insoluble ash, percent by mass, *Max*	0.2	**A-5** of IS 7928
v)	Protein (N × 5.7), percent by mass, *Max*	7.0	IS 7219
vi)	Acid insoluble matter, percent by mass, *Max*	2.0	Annex D
vii)	Viscosity of 1 percent solution, mPas, *Max*	500	Annex F
viii)	Fat, percent by mass, *Max*	1	IS/ISO 6492
ix)	Anthraquinones, mg/kg, *Max*	0.5	Annex G
x)	Lead (as Pb), mg/kg, *Max*	2.0	**15** of IS 1699
xi)	Arsenic (as As), mg/kg, *Max*	1.0	**15** of IS 1699
xii)	Heavy metals (as Pb), mg/kg, *Max*	40	**16** of IS 1699

ANNEX A

(*Clause* 3.2.3)

TEST FOR FORMATION OF GEL WITH XANTHAN GUM

A-1 Weigh 1.5 g of the sample and 1.5 g of xanthan gum and blend them. Add this blend with rapid stirring into 300 ml water at 80°C in a 400 ml beaker. Stir until the mixture is dissolved and continue stirring for an extra 30 min after dissolution (maintain the temperature above 60°C during the stirring process). Discontinue stirring and allow the mixture to cool at room temperature for at least 2 h. A firm, viscoelastic gel forms after the temperature drops below 40°C. No such gel forms in a 1 percent control solution of cassia gum or xanthan gum alone prepared in a similar manner.

ANNEX B

(*Clause* 3.2.4)

IDENTIFICATION OF SUGARS

B-1 Boil mixture of 100 mg of the samples and 20 ml of 10 percent sulphuric acid for 3 h. Allow to cool and add excess barium carbonate, mixing with a magnetic stirrer until the solution is of *p*H 7, and filter. Evaporate the filtrate in a rotary evaporator at 30-50°C in vacuum until a crystalline (or syrupy) residue is obtained. Dissolve in 10 ml of 40 percent methanol. This is the hydrolysate. Place 1 to 10 microlitre spots of hydrolysates on the starting line of two chromatoplates and spots containing 1 to 10 micrograms of galactose and mannose expected to be present in the hydrolysate.

B-2 Use two solvent systems one for each plate to develop the plates as follows:

a) A mixture of formic acid, methyl ethyl ketone, tertiary butanol and water (15 : 30 : 40 : 15 by volume); and

b) A mixture of isopropanol, pyridine, acetic acid and water (40 : 40 : 5 : 20 by volume).

B-3 After development, spray with a solution of 1.23 g anisidine and 1.66 g phthalic acid in 100 ml ethanol and heat the plates at 100°C for 10 min. A greenish yellow colour is produced with hexoses, a red colour with pentoses and a brown colour with uronic acids. Compare sample spots with those for the solution of galactose and mannose.

ANNEX C

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF GALACTOMANNANS

C-1 Calculate galactomanans, percent by mass as follow:

100 – Percent [Acid insoluble matter[1] + Total ash[1] + Loss on drying[1] + Protein[1]]

---

[1] As calculated by the methods given in col 4 of Table 1.

ANNEX D

[*Table* 1, *Sl No.* (vi)]

DETERMINATION OF ACID INSOLUBLE MATTER

D-1 Transfer 1.5 g of the sample, accurately weighed, into a 250 ml beaker containing 150 ml of water and 15 ml of 1 percent sulphuric acid. Cover the beaker with a watch glass and heat the mixture on a steam bath for 6 h rubbing down the wall of the beaker frequently with a rubber-tipped stirring rod and replacing any water lost by evaporation. At the end of the 6 h heating period add about 500 mg of a suitable filter aid, accurately weighed, and filter through a tared Gooch crucible provided with an asbestos pad. Wash the residue several times with hot water, dry the crucible and its contents at 105°C for 3 h cool in a desiccator and weigh.

D-2 Calculate the percent of acid insoluble matter as follows:

$$\text{Acid insoluble matter, percent by mass} = \frac{W_3 - W_2}{W_1} \times 100$$

where

W_1 = weight of sample taken,
W_2 = weight of empty Gooch crucible, and
W_3 = weight of Gooch crucible with insoluble contents.

ANNEX E

(*Clause* 3.5)

DETERMINATION OF RESIDUAL ISOPROPANOL BY GAS CHROMATOGRAPHY

E-1 EQUIPMENT

E-1.1 Gas Chromatography Conditions

a) Column: 25 percent Diphenyl – 75 percent dimethylpolysiloxane (60 m × 0.25 mm i.d., 0.25 ìm film) [Aquatic-2 (GL-Sciences Inc.) or equivalent]
b) Carrier gas: Helium
c) Flow rate: 1.5 ml/min
d) Detector: Flame-ionization detector (FID)
e) Temperatures:
    1) Injector: 280° C
    2) Column: Hold for 6 min at 40°, then

40-110 °C at 4 °C/min, 110-250 °C at 25 °C/min, hold for 10 min at 250 °C- detector: 250 °C

E-1.2 Analytical Balance, least count = 0.000 1 g.

E-1.3 Wrist Action Mechanical Shaker

E-2 REAGENTS

E-2.1 Isopropanol, Chromatography Grade

E-2.2 Tertiary Butyl Alcohol, Chromatography Grade

E-2.3 Double Distilled Water

E-2.4 Standard Solutions

E-2.4.1 *Isopropanol Standard Solution* — Transfer 100 mg each of chromatography grade isopropanol into a 100 ml volumetric flask containing about 90 ml water and dilute to 100 ml with water.

E-2.4.2 *TBA Standard Solution* — Transfer 100 mg of chromatography grade tertiary-butyl alcohol (TBA) into a 100 ml volumetric flask containing about 90 ml water and dilute to 100 ml with water.

Mixed standard solutions: Transfer 1, 2, 3, 4 and 5 ml of Isopropanol standard solution into each of five 100 ml volumetric flasks. Add 4 ml of TBA standard solution to each flask and dilute to volume with water.

E-3 SAMPLE PREPARATION

Disperse 1 ml of a suitable antifoam emulsion in 200 ml of water contained in a 1 000 ml 24/40 round-bottom distilling flask. Add about 4 g of the sample, accurately weighed, and shake for 1 h on a wrist-action mechanical shaker. Connect the flask to a fractionating column, and distil about 95 ml, adjusting the heat so that foam does not enter the column. Add 4 ml of TBA standard solution to the distillate and make up to 100 ml with water to obtain the sample solution.

E-4 STANDARD CURVES

Inject 1 µl of each standard solution into the chromatograph. Measure the peak areas for isopropanol and TBA. Construct the standard curves by plotting the ratios of the peak areas of each of the isopropanols/ TBA against the concentrations of each isopropanol (mg/ml) in the mixed standard solutions.

E-5 PROCEDURE

Inject 1 µl of the sample solution into the chromatograph. Measure the peak areas for isopropanol and TBA. Calculate the ratios of the peak areas of each isopropanol/TBA, and obtain the concentration of each isopropanol from the standard curves.

Calculate the percentage of isopropanol from:

Percent isopropanol = $(C \times 100/W \times 1\,000) \times 100$

where

C = concentration of isopropanol, mg/ml, and

W = weight of sample, g.

ANNEX F

[*Table* 1, *Sl No.* (vii)]

DETERMINATION OF VISCOSITY

F-1 EQUIPMENTS

F-1.1 Brookfield Viscometer, type A with RVT Spindle 1with rotational frequency of 20/min [*see* IS 13360 (Part 11/Sec 10)].

F-1.2 Magnetic Stirrer, with heating and thermal regulation facility.

F-1.3 Water Bath

F-1.4 Analytical Balance, least count of 0.1 g.

F-2 PROCEDURE

F-2.1 Weigh 5 g of the sample in a plastic dish. Weigh 495 g of distilled water at 20 °C in a 1 000 ml beaker.

F-2.2 Add a magnetic bar and place the beaker on the magnetic stirrer. Adjust the speed of agitation to 750 rpm. Introduce quickly the 5 g of sample in the water and cover the beaker with a watch glass. Keep the temperature at 90° C for 15 min.

F-2.3 Cool the solution at 25° C (the cooling must be ± 1.5° C) in a water bath and measure the viscosity after 2 h at 25° C using a RVT Brookfield Spindle 1, speed 20 rpm as per the method given in IS 13360 (Part 11 /Sec 10).

ANNEX G

[*Table* 1, *Sl No*. (ix)]

DETERMINATION OF ANTHRAQUINONES

G-1 EQUIPMENTS

G-1.1 High Performance Liquid Chromatograph (HPLC), with photodiode array detector maintainable at 435nm

G-1.2 HPLC Column — Hypersil C18 (250 mm × 4.6 mm ID, 5 µm) or equivalent

G-1.3 Analytical Balance, with least count of 0.001g

G-1.4 Micro Syringe, with capacity of 100 µl and least count of 1 µl.

G-1.5 Ultrasound Mixing Bath

G-1.6 Extrulet Column, previously neutralized with a *p*H 9.0 buffer.

G-2 REAGENTS

G-2.1 1,8-dihydroxy-3-methoxy-6-methyl-anthraquinone, of known purity.

G-2.2 1,8-dihydroxy Anthroquinone, of known purity.

G-2.3 Methanol, HPLC grade.

G-2.4 Trifluoroacetic Acid, HPLC grade.

G-2.5 Acetonitrile, HPLC grade.

G-2.6 9.0 *p*H buffer solution .

G-3 STANDARD SOLUTIONS

G-3.1 Stock standard solutions (100 mg/l): For each of the specific anthraquinone standards (1,8- dihydroxy-3-methoxy-6-methyl-anthraquinone) and for the internal standard (1,8-dihydroxy anthroquinone), accurately weigh about 1 mg (±0.01 mg) of the standard. Transfer to 10 ml volumetric flasks with about 5 ml of methanol, sonicate for 15 min and dilute to volume with methanol. Store these solutions in amber coloured bottles at 4°C (the solutions are stable for 2 weeks under these conditions).

G-3.2 Mixed standard solution (10 mg/l): Pipette 1 ml of each of the specific anthraquinone stock standard solutions into a 10 ml volumetric flask and dilute to volume with methanol.

G-3.3 Working standard solutions: To each of five 10 ml volumetric flask pipette 5, 2, 1, 0.5 and 0 ml respectively of the Mixed standard solution, pipette 1 ml of the Internal standard stock solution to each flask, mix and dilute to volume with methanol.

G-4 SAMPLE PREPARATION

G-4.1 Accurately weigh about 0.40 g of the sample into a 50 ml roundbottom flask. Add 20 ml trifluoroacetic acid and reflux at 70°C for 4 h. Cool the sample to ambient temperature and evaporate to dryness using a rotary evaporator. Add 3 ml of acetonitrile/ $NaHCO_3$ (0.2 percent) [60:40 (*v/v*)] and sonicate for 30 min.

G-4.2 Transfer the solution in a centrifuge tube and run it at 5 000 rpm for 30 min. Filter the supernatant solution through an Extrulet column previously neutralized with a *p*H 9.0 buffer. Pipette 900 µl of this filtered sample solution into a 2.5 ml volume vial and add 100 µl of the internal standard stock solution and mix thoroughly.

G-5 PROCEDURE

G-5.1 Chromatographic conditions:

Column: Hypersil C18 (250 mm × 4.6 mm ID, 5 µm) or equivalent

Mobile phase:

a) 0.1 percent trifluoroacetic acid in water, and
b) Acetonitrile (HPLC grade).

Injection volume	:	50 µl
Run time	:	60 min
Flow rate	:	1 ml/min
Detector	:	photodiode array detector. Quantification is performed at 435 nm

G-5.2 Standard curves: Inject 50 µl of each working standard solution and internal standard solution. Construct the standard curves by plotting the ratios of the peak areas of each of the specific anthraquinone / internal standard against the concentrations of each working standard solution (mg/l).

G-5.3 Inject 50 µl of the sample solution and the internal standard solution.

Calculate the ratios of the peak areas of each specific anthraquinone/internal standard, and obtain the concentration (C) of each specific anthraquinone from the standard curves.

Calculate the percentage of each specific anthraquinone from:

$$\text{Anthraquinone (mg/kg)} = C \times 3 \times 1\,000 / (100 \times 0.9 \times W)$$

where

C = concentration of specific anthraquinone, mg/l, and
W = weight of sample, g.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publications), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

This Indian Standard has been developed from Doc No.: FAD 08 (2920).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.org.in

Regional Offices:		*Telephones*
Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	2323 7617 2323 3841
Eastern	: 1/14 C.I.T. Scheme VII M, V. I. P. Road, Kankurgachi KOLKATA 700054	2337 8499, 2337 8561 2337 8626, 2337 9120
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg, CHANDIGARH 160019	26 50206 265 0290
Southern	: C.I.T. Campus, IV Cross Road, CHENNAI 600113	2254 1216, 2254 1442 2254 2519, 2254 2315
Western	: Manakalaya, E9 MIDC, Marol, Andheri (East) MUMBAI 400093	2832 9295, 2832 7858 2832 7891, 2832 7892

Branches: AHMEDABAD. BENGALURU. BHOPAL. BHUBANESHWAR. COIMBATORE. DEHRADUN. DURGAPUR. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. JAMMU. JAMSHEDPUR. KOCHI. LUCKNOW. NAGPUR. PARWANOO. PATNA. PUNE. RAIPUR. RAJKOT. VISAKHAPATNAM.

Published by BIS, New Delhi